

INVESTIGATING EFFICIENCY OF GLUCOSE-DERIVED GRAPHENE QUANTUM DOTS IN PHOTOCATALYSIS

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(2K21/MSCPHY/05)

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ABSTRACT

Graphene is unique in that it has a zero bandgap, unlike graphene quantum dots. However, the functionalization of graphene allows a significant alteration in its properties. Here, graphene quantum dots (GQDs) were fabricated in an aqueous medium via carbonization, using glucose as a carbon precursor at ambient temperature. The process is environmentally benign and cost-effective with de-ionized water as a solvent and ammonia as a catalyst. The chemical compositional and morphological analysis of GQDs was examined by FTIR and HRTEM, respectively. The as-synthesized GQDs were spherical and showed an absorption peak at 280 nm. Additionally, they exhibit strong emission in the blue region at 350 nm and display excitation wavelength-dependent features. The resulting GQDs have a bandgap of 3.81 eV. The study evaluated the catalytic activity of GQDs under NaBH_4 mediated photoreduction and compared their efficiency in degrading the organic rhodamine B dye under visible and UV light for 16 minutes. The outcome of the study revealed that GQDs possess exceptional photocatalytic activity.

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LIST OF SYMBOLS AND ABBREVIATIONS

NPs	Nanoparticles
GQD	Graphene quantum dot
GO	Graphene oxide
GNP	Graphene nanoplatelets
GNR	Graphene nanoribbon
rGO	Reduced graphene oxide
UV-Vis	Ultraviolet-Visible
NIR	Near-infrared
PL	Photoluminescence
FTIR	Fourier transform infrared spectrometer
TEM	Transmission electron microscope
HRTEM	High-resolution transmission electron microscopy
RhB	Rhodamine B
ROS	Reactive oxygen species

CHAPTER 1

INTRODUCTION

1.1 Nanotechnology

Nanotechnology is a rapidly advancing domain of science and engineering that deals with the design, synthesis, manipulation, and characterization of materials at the nanoscale level. Nanotechnology involves the study of materials with dimensions ranging from 1 to 100 nanometers, which is roughly 1000 times smaller than the diameter of a human hair [1]. At the nanoscale, materials exhibit unique properties that are not observed in bulk materials due to the high surface-to-volume ratio, quantum confinement, and other quantum mechanical effects [2]. These properties have opened up exciting possibilities for the advancement of innovative materials, devices, and utilizations across diverse domains such as electronics, medicine, energy, and environmental science.

Nanotechnology encompasses various disciplines, including physics, chemistry, materials science, biology, and engineering. Scientists and researchers in this field work with nanomaterials and nanodevices to understand their unique properties and develop new applications. One of the fundamental concepts in nanotechnology is that material at the nanoscale often exhibit different properties and behaviors compared to the same materials at larger scales. This is due to the increased surface area, quantum effects, and other factors that become more pronounced at the nanoscale. Nanotechnology holds the

capacity to revamp many areas, including electronics, medicine, energy, environmental science, and materials science.

1.2 Literature review

1.2.1 Different Forms of Graphene:

Graphene is a distinctive manifestation of carbon possessing an innovative structural configuration, initially unveiled in 2004. It is an unprecedented two-dimensional material that comprises densely packed carbon atoms in a flat sheet exhibiting a hexagonal lattice pattern [3]. It offers unique physicochemical qualities such as an expansive surface area, outstanding biological compatibility, great mechanical strength, excellent thermal conductivity, and quick electron movement, making it adaptable to different applications [4]. Although graphene has various uses, it cannot be employed in many of them owing to the lack of energy gap in graphene, low water dispersivity, and low spectrum absorption [5], [6]. The enriching of chemical functional groups to the surface of graphene through functionalization can lead to a modification in graphene's thermal and electrical conductivity. Graphene comes in many different forms, including graphene oxide (GO), graphene nanoplatelets (GNPs), graphene nanoribbons (GNRs), reduced graphene oxide (rGO), and graphene quantum dots (GQDs). Functionalizing GO with groups like carbonyl, hydroxyl, and epoxy can increase its hydrophilicity, making it a better choice for solvent-based samples than pristine graphene [7].

These are just a few examples of the different forms of graphene that have been synthesized and studied. As research and development in graphene continue, new forms and derivatives with unique properties are likely to emerge, further expanding the range of applications for this extraordinary material.

1.2.2 Graphene Quantum Dots:

Graphene quantum dots are nanoscale structures composed of graphene, a monoatomic layer of carbon atoms organized in a planar lattice (shown in Fig. 1.1). When the size of graphene is reduced to a nanoscale level, typically less than 10 nanometers, it exhibits quantum confinement effects, leading to the generation of graphene quantum dots. [8,9]. The quantum confinement effect leads to the encapsulation of charge carriers (electrons and holes) within a compact space. This spatial limitation constrains the mobility of electrons, resulting in discrete energy levels known as energy "states" or "levels." The energy spacing between these levels varies based on the size and configuration of the quantum dot. The majority of GQDs are round or elliptical in structure, depending on the fabrication method employed [10]. Due to their compact dimensions and elevated surface-to-volume ratio, they are unique and diverse. Solar cells, biosensors, light-emitting diodes, and photodetectors represent a few of the potential implementations. They are excellent for fluorescence sensing, photodegradation of dyes, and bioimaging applications due to their exceptional optical capabilities [11].

In addition to GO, GQDs are also attractive due to their excellent water solubility and chemical robustness, which make them non-toxic, highly stable, and biocompatible. The size-dependent opening of the bandgap is a crucial factor that distinguishes GQDs from graphene, setting clear boundaries between the two materials [5,12]. They may also be mixed with other substances to create GQDs-based nanomaterials to establish collaborative effects to promote catalytic interactions with target analytes in many contexts. Also, GQDs-based nanocomposites have several uses in electrochemical sensors [13].

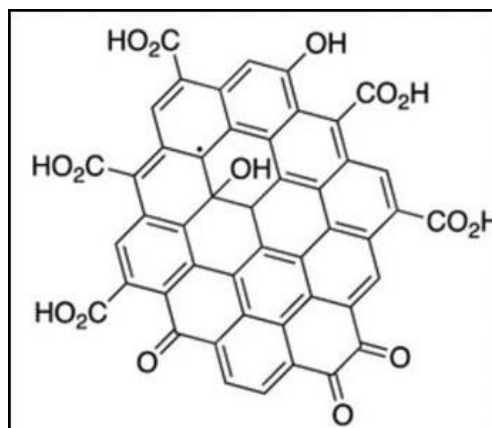


Fig. 1.1 Structure of graphene quantum dots

1.2.3 Key properties of graphene quantum dots

Graphene quantum dots possess several important properties that contribute to their significant scientific and technological potential. Here are some key properties of GQDs:

(i) Quantum Confinement: GQDs demonstrate the phenomenon of quantum confinement effects due to their nanoscale dimensions. This confinement leads to discrete energy levels and tunable electronic properties, making GQDs suitable for applications in quantum computing and electronic devices.

(ii) Optical Properties: GQDs demonstrate strong photoluminescence properties. They can absorb and emit light across a broad spectrum of wavelengths, from the UV spectrum to infrared range, making them valuable for optoelectronic devices, light-emitting diodes (LEDs), solar cells, and bioimaging.

(iii) High Surface-to-Volume Ratio: GQDs have a high surface-to-volume ratio due to their small size and two-dimensional structure. This property enhances their interaction with other materials and facilitates efficient charge transfer and catalytic reactions. It makes GQDs useful in energy storage devices, catalysis, and sensing applications.

(iv) Electrical Conductivity: GQDs possess high electrical conductivity, stemming from the intrinsic conductivity of graphene. This property enables their application in conductive inks, transparent conductive films, and electronic devices.

1.2.4 Applications

GQDs possess unique electronic, optical, and chemical properties, positioning them as a promising candidate for various applications. Here are some of the potential applications of GQDs:

(i) Optoelectronics and Photonics: GQDs exhibit size-dependent photoluminescence, meaning their emission wavelength can be tuned by controlling their size. This characteristic renders them highly appropriate for implementation in optoelectronic devices like light-emitting diodes (LEDs), solar cells, and photodetectors.

(ii) Biological Imaging and Sensing: GQDs have excellent biocompatibility, low toxicity, and high photostability, rendering them suitable for biological imaging and sensing applications. They have the capability to serve as fluorescent probes for imaging purposes and tracking biological molecules, cells, and tissues. GQDs can also act as efficient sensors for detecting biomolecules, such as DNA, proteins, and heavy metal ions.

(iii) Energy Storage and Conversion: The exceptional electrochemical properties, excellent electrical conductivity, and high specific surface area of GQDs enable their effective utilization in energy storage devices, such as supercapacitors and batteries. They can enhance the performance of these devices by improving charge storage capacity, cycling stability, and rate capability. GQDs can also be employed as catalysts or co-catalysts for electrochemical reactions in energy conversion devices like fuel cells and water-splitting systems.

(iv) Environmental Applications: GQDs have shown promise in environmental applications such as water purification and pollutant detection. Their large surface area and unique surface chemistry enable efficient adsorption of organic pollutants, heavy metal ions, and dyes from water. GQDs can also be integrated into sensing platforms for detecting and monitoring environmental pollutants.

(v) Electronic and Sensing Devices: GQDs can be used in electronic devices like field-effect transistors (FETs) and sensors. They exhibit high charge carrier mobility and tunable electrical properties, making them suitable for flexible electronics and wearable devices. GQDs can also be incorporated into gas sensors, biosensors, and chemical sensors for detecting a wide range of analytes.

(vi) Catalysis: GQDs have shown promising catalytic activity for various chemical reactions, including oxygen reduction reaction (ORR), hydrogen evolution reaction (HER), and photocatalysis. Their unique surface properties and abundant edge sites make them efficient catalysts for improving reaction rates and selectivity.

1.3 Aim and Scope of the Study

- Green synthesis of aqueous graphene quantum dots using glucose powder
- Analyzing the developed GQDs through various characterization techniques.
- Investigating the photocatalytic activity of GQDs in degradation of dyes.

CHAPTER 2

MATERIALS AND METHODS

2.1 Synthesis Methods of Nanoparticles:

In the field of nanoscience, diverse physical and chemical traits of nanomaterials are often examined via construction approaches. Depending on the starting material, NPs preparation techniques developed in the past few years are separated into two groups, namely top-down and bottom-up methodologies [14].

The top-down approach employs initial macroscopic structures as a starting point. This method commences with larger particles and subsequently subjects them to a series of operations, gradually reducing their size to nanoparticles [15,16]. This involves the hydrothermal process, chemical oxidation method, chemical vapor deposition, electrochemical oxidation method, pulsed laser ablation technique, ultrasonic aided method, and also a combination of these [17]. Bottom-up approaches to nanomaterial production involve the reduction of material constituents to the atomic level, accompanied by subsequent processes that facilitate the creation of nanostructures [18]. Carbonization, microwave irradiation, and electron beam irradiation are some of the technologies available [17]. Because top-down synthesis processes include machine cost complexity, surface imperfections, contamination, cost and complexity of clean rooms, material damage, and heat dissipation. Bottom-up procedures are becoming an increasingly significant addition to top-down strategies.

There are three main approaches to nanoparticle production, each chosen based on specific needs and considerations. Each method has its own set of pros and cons, with the selection of a production method typically dependent on the availability of facilities [4]. Physical methods are well-suited for limited-scale production, while chemical methods are preferred when cost efficiency is a primary concern. Biological methods, on the other hand, hold distinct significance in nanoparticle synthesis [4,8].

1. Physical Methods
2. Chemical Methods
3. Biological Methods

2.1.1 Physical Methods:

Physical methods are based on physical principles and processes for the fabrication of nanoparticles. These methods involve the conversion of bulk materials into nanoparticles through mechanical, thermal, or electrical means. In this approach, nanoparticles are significantly influenced by factors such as drilling duration, medium composition, and initial technique. It is a typical depiction of the top-down method [19]. Some common physical methods include:

(i) Mechanical Milling: This technique employs mechanical forces, such as ball milling or attrition, to decrease the dimensions of bulk materials and produce nanoparticles. It entails grinding or crushing the material in a high-energy mill, resulting in the reduction of particle size. The speed and duration of the milling process are crucial factors in the synthesis of nanoparticles with the desired size [5,11].

(ii) Laser Ablation: Laser ablation is a technique that employs laser pulses to vaporize a specific material, leading to the creation of nanoparticles. Subsequently, the vaporized

substance undergoes condensation and solidification, ultimately forming nanoparticles [12].

2.1.2 Chemical Synthesis:

Chemical synthesis is a widely employed method for producing nanoparticles. It involves the formation of nanoparticles through chemical reactions, often using a precursor solution or a mixture of reactants. This method embodies the principles of a bottom-up approach [20]. The reaction conditions, such as temperature, pressure, and concentration, are precisely regulated to attain the intended dimensions and composition of nanoparticles. Chemical synthesis enables meticulous regulation of particle dimensions, morphology and surface properties [21]. Some common techniques within chemical synthesis include:

(i) Sol-Gel: The method described involves creating nanoparticles by hydrolyzing and condensing precursor molecules within a solution or gel matrix. This approach enables meticulous control over the size and morphology of nanoparticles.

(ii) Hydrothermal synthesis: It is among the most frequently employed methodologies for nanoparticle fabrication, predominantly relying on chemical reactions. Hydrothermal synthesis is a versatile technique that enables nanoparticle synthesis across a broad temperature range, including room temperature and extremely high temperatures. This method offers numerous advantages compared to physical and biological approaches. However, it is important to note that the nanomaterials produced through hydrothermal synthesis may exhibit instability at elevated temperature ranges [32].

2.1.3 Biological Methods:

Biological methods utilize living organisms or their components to produce nanoparticles. These methods often involve the use of microorganisms, plants, or

biological templates to synthesize and assemble nanoparticles [22]. Some common biological methods include:

(i) *Microorganism-mediated Synthesis:* Microorganisms, including bacteria and fungi, possess the capability to produce nanoparticles by reducing metal ions present in a solution. This reduction process leads to the formation of nanoparticles. Essentially, these microorganisms function as bioreactors that facilitate the production of nanoparticles.

(ii) *Plant-mediated Synthesis:* Living plants or plant extracts have the potential to generate nanoparticles through the utilization of their natural bioactive compounds. These plant extracts function as agents that both reduce and stabilize the nanoparticles, thereby enabling their formation.

CHAPTER 3

CHARACTERIZATION TECHNIQUES

In nanotechnology, characterization techniques are crucial for analyzing and understanding the properties, structure, and behaviour of nanoscale materials and structures. These techniques allow scientists and engineers to examine nanomaterials and determine their composition, size, shape, surface properties, and other important characteristics. Here are some commonly used characterization techniques in nanotechnology.

3.1 High-resolution transmission electron microscope (HRTEM)

HRTEM is a potent technique that involves transmitting a stream of electrons through a thin sample and provides detailed information about the sample's internal structure, such as crystal structure, lattice defects, and atomic arrangements. TALOS (Transmission Electron Aberration-corrected Low Voltage System) is a popular model of transmission electron microscope (TEM) commonly used for HRTEM imaging, as shown in Fig. 3.1.



Fig. 3.1 HRTEM using TALOS

3.2 Fourier Transform infrared spectrometer (FTIR)

The Perkin Elmer Spectrum Two FTIR spectrometer, as shown in Fig. 3.2, was utilized to capture the FTIR spectrum by detecting the absorption or transmission of infrared light through the sample. It identifies chemical functional groups present in the material, providing information about its molecular composition and bonding.



Fig. 3.2 Spectrum Two FTIR spectrometer

3.3 UV-Visible-NIR spectrometer

The Perkin Elmer Lambda 750 UV/Vis/NIR spectrometer was employed to obtain the UV-Vis absorption spectra of the samples within the 200-600 nm range, shown in Fig. 3.3. PerkinElmer Lambda 750 includes a double-beam optical design, a high-energy deuterium and tungsten light source, a monochromator, and a variety of measurement modes such as absorbance, transmittance, reflectance, and kinetics.



Fig. 3.3 UV/Vis/NIR spectrometer (Model: PerkinElmer LambdaTM 750)

3.4 Spectrofluorometer

The Horiba Jobin Yvon Fluorolog 3 is a highly regarded spectrofluorometer model produced by Horiba Jobin Yvon, a prominent manufacturer of scientific instruments. It is designed for advanced fluorescence measurements and offers a range of features and capabilities. The measurement of the photoluminescence (PL) and PL-excitation spectra were conducted using a spectrofluorometer (Horiba Jobin Yvon, Fluorolog-3), which was fitted with a 450 W xenon lamp and a photomultiplier tube as shown in Fig. 3.4.



Fig. 3.4 Horiba Jobin Yvon, Fluorolog-3

CHAPTER 4

Investigating efficiency of Glucose-derived Graphene Quantum Dots in Photocatalysis

4.1 Experimental setup

4.1.1 Materials used:

D-(+)-Glucose (99.5%) was procured from Sigma-Aldrich. Ammonia solution (NH₄OH) 25% and sodium hydroxide (NaOH) pellets (97%) were acquired from Rankem. The materials used throughout the experiment were conventional and used without additional purification. Throughout the experiments, ultrapure water was employed as a solvent, which had a resistivity coefficient of 18.2 MΩ-cm.

4.1.2 Synthesis of graphene quantum dots:

Initially, 2 g of glucose was heated in a beaker on a hot plate at 200 °C. After 4-5 minutes of heating, the powdered glucose turned into liquid form. The liquid was then heated for another 15 minutes and thus the color was altered from colorless to yellow-brownish. Further, prepared a 12% ammonia solution in 100 ml of ultrapure water. Successively, added the orange solution, gradually, to the prepared ammonia solution with rapid stirring. The final solution was subjected to thermal treatment at 70 °C for 4 hours until the solution has adjusted to pH 7 and the ammonia odor had evaporated.

Fig 4.1 represents the process of synthesizing graphene quantum dots. In carbonization, ammonia dehydrates the glucose molecules in an aqueous solution. During the generation of GQDs, glucose molecules' hydrogen atoms communicate through the hydroxyl groups of a neighboring glucose monohydrate. Due to the generation of water molecules, carbon atoms create covalent bonds with one another, which results in the creation of graphene quantum dots [23]. The material attributes are presented in Table 4.1.

Table 4.1 Physical properties of Glucose

Chemical formula	$C_6H_{12}O_6$
Molecular weight	180.16 g/mol
Appearance	White powder
Melting point	146 °C

4.1.3 Preparation of samples for photocatalysis:

A stock solution of 50 μ M concentration of rhodamine dye of molar mass given in Table 4.2. was prepared by adding 1.1 mg of the dye to 50 mL of deionized water. This was further diluted to 10 μ M. The mixture was agitated for 30 minutes to validate the thorough blending of the dye. Subsequently, the dye solution was relocated to a pair of quartz cells, with one cell being subjected to UV light and the other exposed to visible light.

Table 4.2 Properties of Rhodamine B

Chemical formula	$C_{28}H_{31}ClN_2O_3$
Molar mass	479.02 g/mol
Appearance	Pink
Solubility in water	8 to 15 g/L (20 °C)

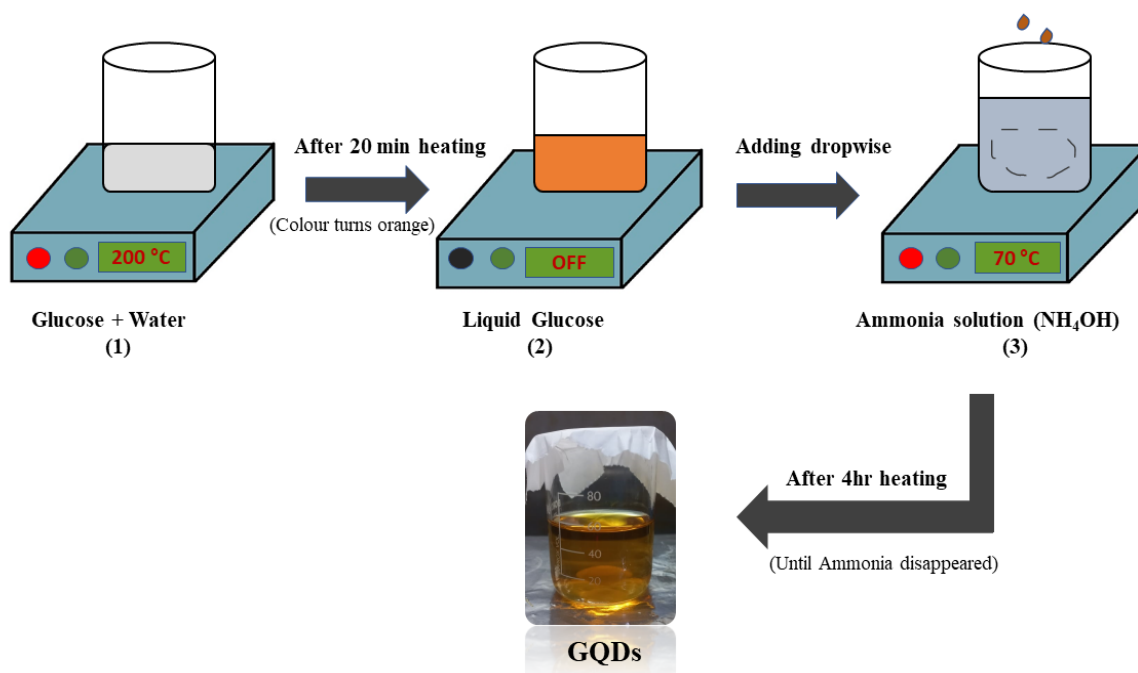


Fig. 4.1 A schematic representation of carbonization for producing graphene quantum dots.

4.2 Results and Discussion

4.2.1 HRTEM Analysis

Figs. 4.2 depicts the HRTEM images at different levels of magnifications along with the particle size analysis of GQDs. Fig. 4.2(a) and (b) show the magnification of as-synthesized GQDs at 50 nm and 10 nm, showing the approximate spherical formations of GQDs. Fig. 4.2(c) represents the range of particle sizes, with the typical particle dimension of 24 nm. In the case of graphene quantum dots, due to their charged nature, they tend to accumulate or aggregate together. The existence of lattice fringes is evidence of the particle's crystallinity, which is visible under certain conditions when the particles are examined with high-resolution imaging techniques.

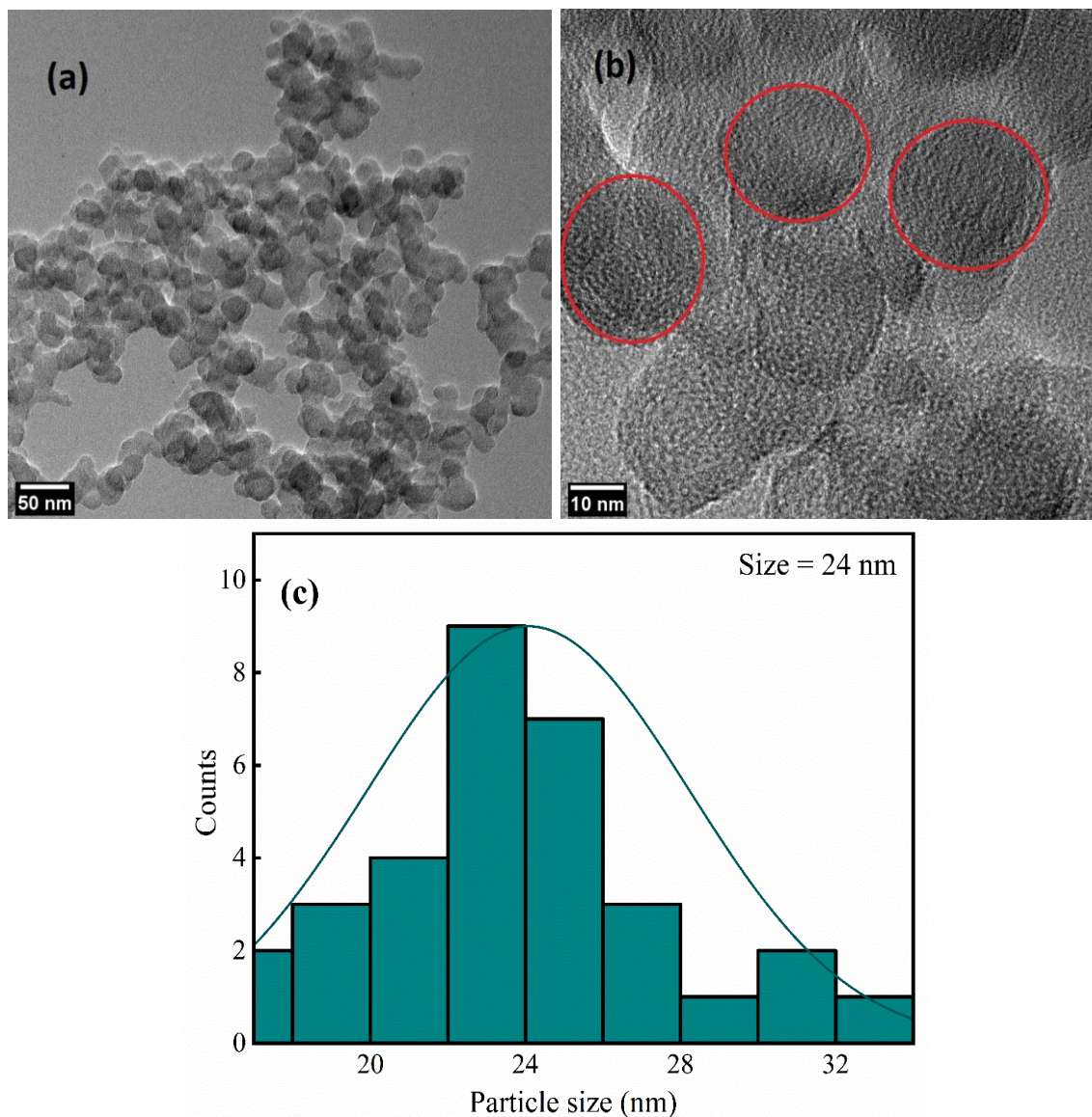


Fig. 4.2 HRTEM images of GQDs at magnifications of 10 nm (a) and 50 nm (b), and particle size distribution of GQDs (c)

4.2.2 FTIR Analysis

Fourier transform analysis is performed to identify and quantify chemical compounds in a sample by analyzing the absorption or transmission of infrared light. Fig. 4.3 displays the FTIR spectrum of GQDs, which verifies the existence of diverse chemical moieties. The broad peak at 3300 cm^{-1} is common to all samples which use water as a solvent and represents the vibrational stretching of the O-H bond. The C=C vibrational stretching

mode, which is the fundamental constituent of GQDs is ascribed to the 1635 cm^{-1} spectral feature. The peaks at 1042 cm^{-1} and 1071 cm^{-1} demonstrate the existence of C-O-H functional groups [24].

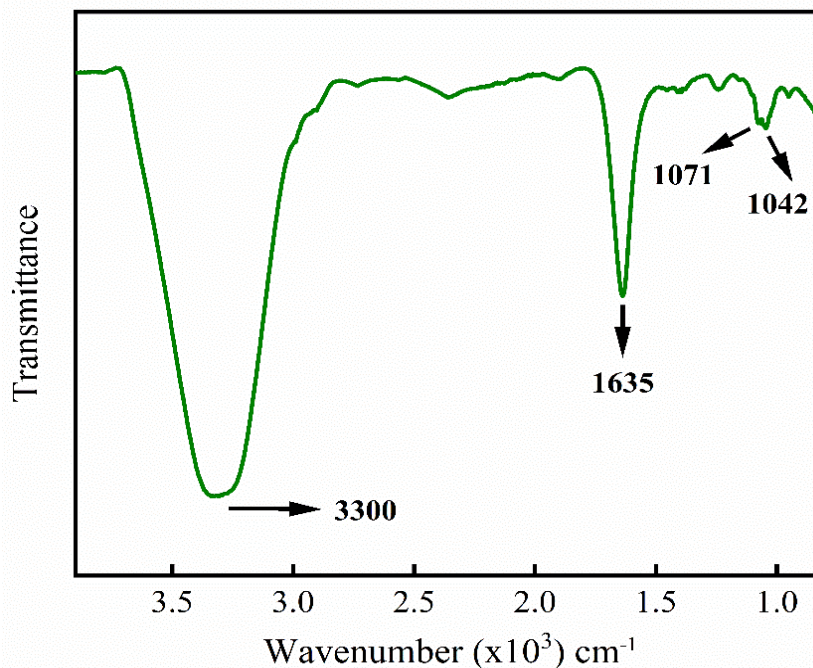


Fig. 4.3 FTIR spectrum of GQDs in water

4.2.3 UV-Vis Absorption Spectra:

The UV-Vis absorption spectrum of as-prepared GQDs within the range 200-600 nm shows two peaks (as shown in Fig. 4.4). The observed transitions correspond to electronic transitions from the π -orbitals to π^* -orbitals and from non-bonding electron pairs to π^* -orbitals. The precursor does not display any significant absorption. The first band maximum at 232 nm is related to the electronic transition of C=C through the π - π^* pathway in the sp^2 region, while another peak is noticed at around 280 nm, which corresponds to the n - π^* transition of C=O in sp^3 hybrid regions that rises due to the existence of oxygen-inclusive heteroatoms in the graphene quantum dot structure [24,26].

These are the main peaks that characterized pure GQDs. No other peaks are observed up to the 600 nm wavelength.

The band gap is a significant property of a material that directly influences its electronic and optical characteristics, including its absorption and emission properties [28]. Therefore, it can be computed based on the absorption parameters by drawing a tangent on the Kubelka-Munk (K-M) function $(\alpha h\nu)^2$ against photon energy $(h\nu)$ plot, commonly termed as the Tauc plot, as illustrated in Fig. 4.4 (inset), where α represents the coefficient of optical absorption, ν is the frequency of light, h refers to the Planck's constant (value equals to 6.626×10^{-34} J.s) [27,28]. The magnitude of the optical band gap (E_g) was determined as 3.81 eV from Tauc's plot, which is consistent with the data already available (displayed in the inset of Fig. 4.4) [29].

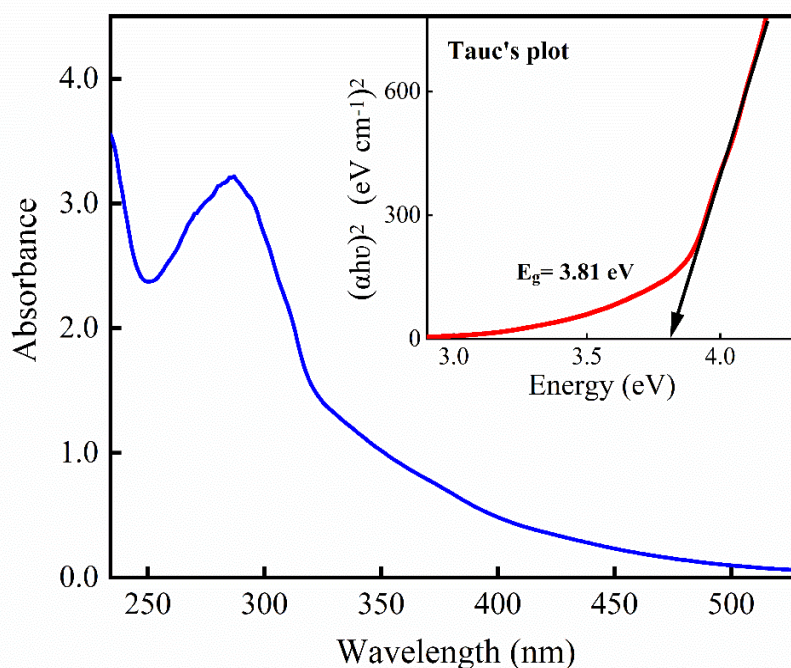


Fig. 4.4 Absorption spectrum of GQDs along with the 'Tauc's plot' analysis (inset)

4.2.4 Photoluminescence of GQDs

The PL and PL-excitation spectra are demonstrated in Figs. 4.5 and 4.6, respectively. The excitation wavelengths for GQDs varied between 350-450 nm. Upon excitation with a 300 nm wavelength, a broad emission peak around 350 nm is observed (as shown in Fig. 4.5). The observed phenomenon shift towards longer wavelengths in the PL peaks and a reduction in the intensity as the excitation increases, is common in carbon-based fluorescent materials, as presented in the visual in Fig. 4.5. Hence, this shows the phenomenon of “excitation-dependent emission”, which is caused by the existence of various dimensions of GQDs. The blue and green emission of GQDs indicates that GQDs have at minimum two distinct emitting states [25]. The PL wavelength of quantum dots varies with particle size, with bigger particles emitting light at longer wavelengths. In our case, strong blue PL is detected in GQDs subjected to irradiation by a 305 nm UV light, as depicted in the subfigure of Fig. 4.7.

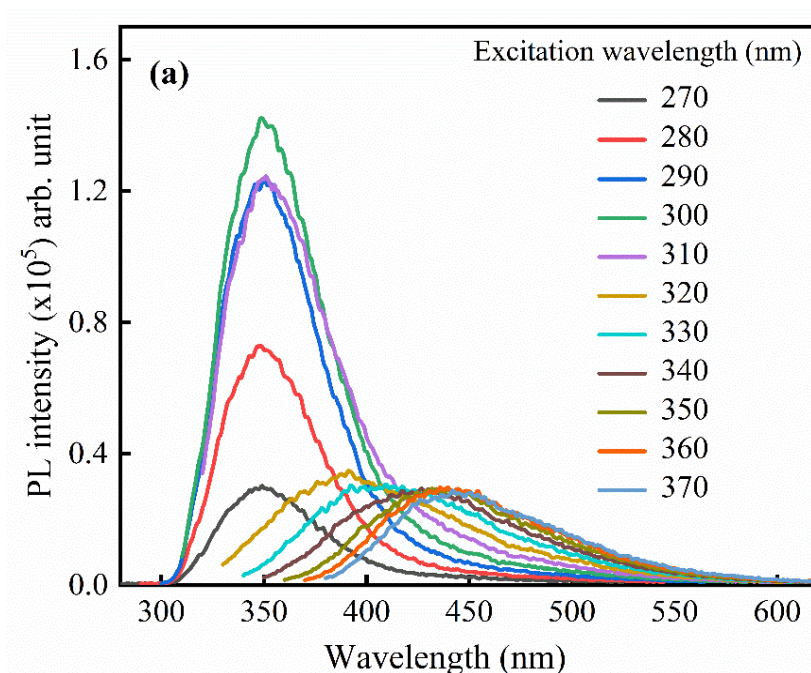


Fig. 4.5 Photoluminescence spectral of GQDs acquired at various excitation wavelengths

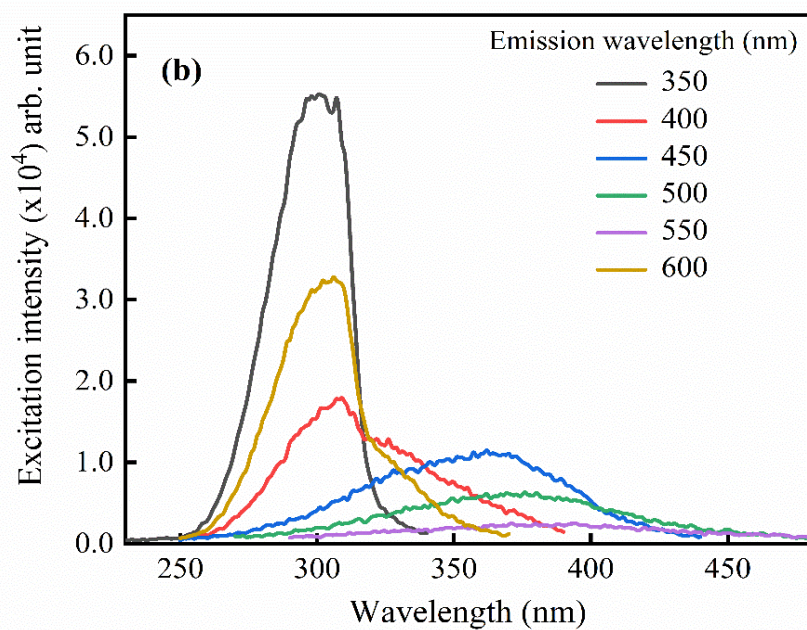


Fig. 4.6 Excitation spectra of GQDs acquired at various emission wavelengths

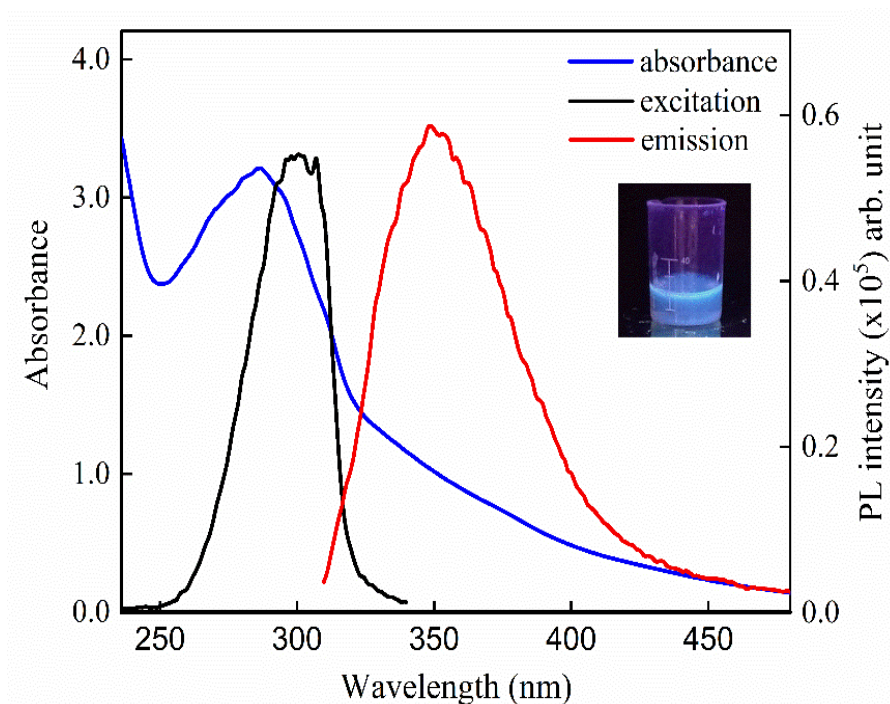


Fig. 4.7 Absorption, PL, and PL-excitation spectra of GQDs along with a photograph showing blue emission under UV irradiation (inset).

4.3 Photocatalytic breakdown of rhodamine B dye

Dyes play a crucial role as coloring agents in the textile industry and find diverse applications such as food additives, tanning, MRI imaging, and more. Unfortunately, a fraction representing 15% of the dyes are inadvertently released into adjacent water bodies throughout their production [30,31]. This discharge has significant detrimental effects on the ecological food web system, impacting both primary and secondary levels, leading to mutagenic disruptions [32]. Even in minuscule quantities, the presence of dyes in water can cause severe environmental issues. In recent years, GQDs have garnered significant attention and emerged as a highly promising and biocompatible zero-dimensional material. A distinct attribute of GQDs is their up-conversion photoluminescence. Up-conversion is just the absorption of multiple low-energy photons as a result of which a single high-energy photon is emitted. GQDs absorb visible light, resulting in the creation of photo-excited electrons and holes. The photogenerated electrons from GQDs were subsequently received by other nanoparticles, aiding the charge carrier separation process. The displaced charged carriers subsequently produced highly reactive radicals, which caused dye photodegradation [23].

The efficiency of GQDs in photocatalytic activity was analyzed for the disintegration of rhodamine B, an organic dye, under the influence of NaBH_4 as a reductant. To examine the degradation percentage of rhodamine B dye, 0.2 mL of GQDs was added as a photocatalyst, and 5 mg of NaBH_4 was added as a reducing agent. The solution was exposed to UV and visible light, and after each exposure, the dye concentration was evaluated by measuring absorption spectra. Rhodamine B is a magenta cationic dye with an absorption band at 554 nm associated with the $n \rightarrow \pi^*$ transition of C=N and C=O groups [33]. Figs. 4.8 illustrate the absorption spectra of rhodamine B dye upon exposure

to UV and visible light at time t . The dye exhibited degradation, transitioning from a pink color to a colorless state within a brief period of 16 minutes. Figs. 4.8(a) and (b) compare the breakdown of rhodamine B dye through UV irradiation in the presence and absence of photocatalysts (GQDs). When both GQDs and NaBH_4 were added, the peak intensity gradually decreases with increased exposure time. The intensity reduction was observed for 16 minutes, and no further changes in color were noticed thereafter. Similarly, NaBH_4 alone exhibited the same mechanism but at a slower rate than the combined effect of GQDs and NaBH_4 . The presence of GQDs + NaBH_4 resulted in the quenching of the absorption band of rhodamine B dye, achieving 86% degradation in about 16 minutes [34,35]. However, in the absence of GQDs, only 30% degradation was observed, highlighting the significant contribution of GQDs in enhancing the rate of degradation [36,37]. Furthermore, as shown in Fig. 4.8(c), the photodegradation of rhodamine B caused by visible light in the presence of GQDs with NaBH_4 was observed to be relatively slow, with the absorption intensity of dye gradually decreasing and reaching 51% degradation in the same time frame. Thus, the degradation rate of the dye under visible light was relatively slower and required more time compared to its degradation under UV irradiation. The results indicate that the GQDs synthesized possess the ability to degrade organic dyes and pollutants, highlighting the importance of further research in this area [31].

To assess the degree of degradation of rhodamine B dye, the percentage degradation R can be calculated using the formula,

$$R = \left(\frac{A_0 - A_t}{A_0} \right) \times 100 \% \quad (4.1)$$

Here, the dye's absorbance was recorded at a wavelength of 554 nm at two different times, with A_0 representing the absorbance at the start of the reaction and A_t representing the

absorbance after a specific time interval t , respectively [37], [38]. The degradation kinetics of the dye were analyzed using a pseudo-first-order chemical reaction model to obtain a rate constant (k) using the equation $\log(A_0/A) = kt$, where A_0 and A represent the initial and subsequent absorbance values of the dye at 554 nm, respectively [40,41]. This assumes that the solvent concentration is greater than the dye concentration. The results showed a direct correlation between $\log(A_0/A)$ and radiation exposure time, indicating that the photodegradation reaction adhered to kinetics that was pseudo-first-order in nature [31,42]. Fig. 4.8(d) illustrates the rate constant (k) values for rhodamine B dye under UV and visible-light exposure. The GQD + NaBH₄ system exhibited a rate constant of 0.126 min⁻¹, whereas NaBH₄ alone resulted in a reaction kinetic constant of 0.021 min⁻¹ under UV irradiation. Conversely, the rate constant for GQD + NaBH₄ was 0.045 min⁻¹ when treated with visible light.

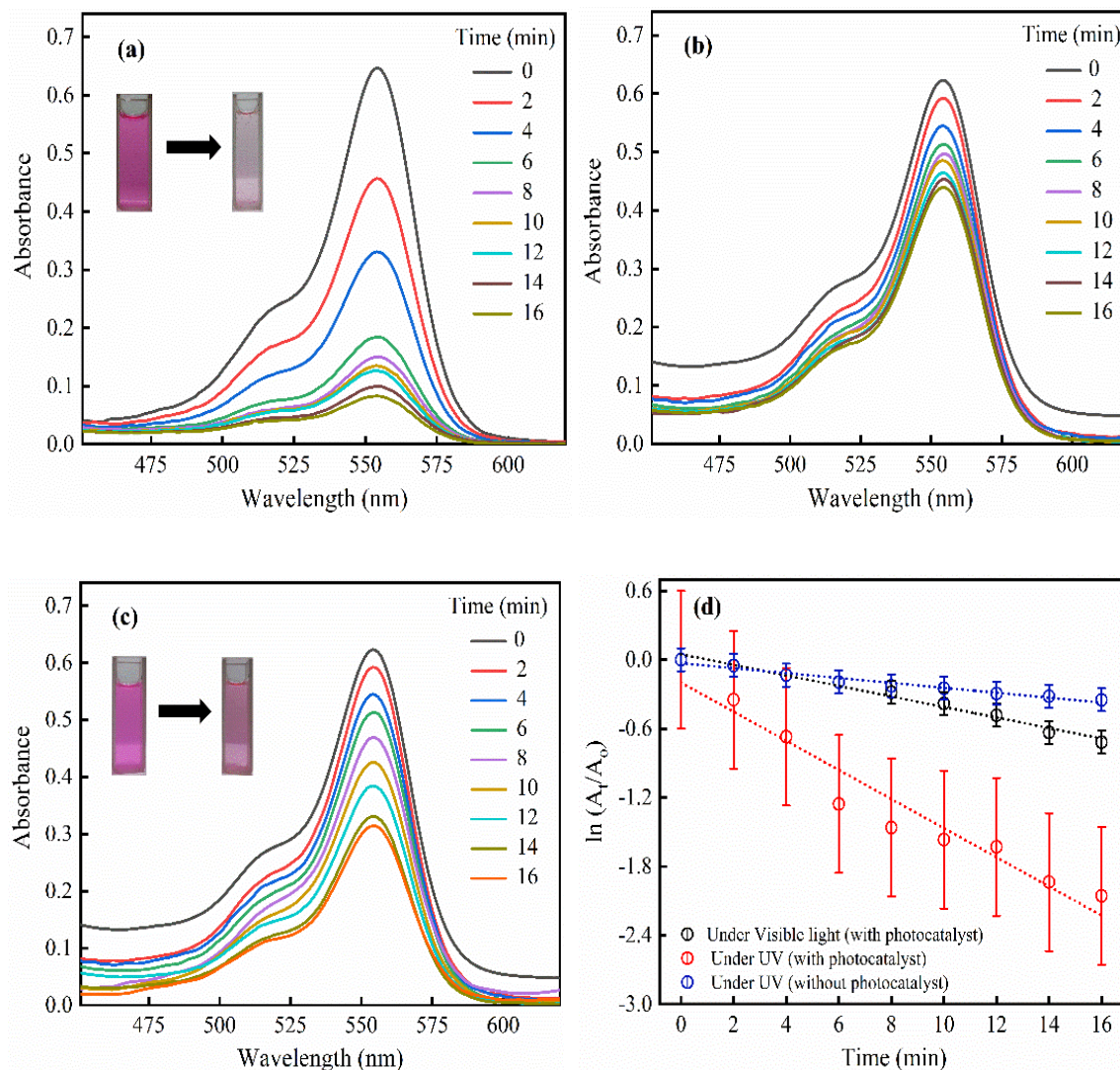


Fig. 4.8 Absorption spectra of rhodamine B with NaBH_4 under UV irradiation (a) with GQDs, (b) without GQDs and (c) without GQDs under visible light. A plot of $\ln(A_t/A_0)$ vs. reaction time (d).

4.4 Degradation mechanism

The photodegradation of rhodamine B by GQDs in the presence of NaBH_4 occurs through a combination of photocatalytic and reduction processes. When exposed to light, GQDs generate reactive oxygen species (ROS), which are powerful oxidizing agents. These ROS can directly degrade rhodamine B by attacking its chromophore, causing it to break down into smaller, less toxic fragments [42]. In addition, NaBH_4 acts as a reducing agent, which

can facilitate the degradation process by donating electrons to rhodamine B, making it more susceptible to oxidation by the ROS generated by GQDs [30]. The presence of both NaBH₄ and GQDs together advances the efficiency of the photodegradation process by increasing the rate of electron transfer between the two components [43]. The adherence of rhodamine B on the GQDs surface also increases the concentration of the dye in the proximity of the ROS generated by GQDs, further promoting its degradation [36,45]. Overall, the mechanism of photodegradation of rhodamine B by GQD in the presence of NaBH₄ involves a combination of photocatalysis and reduction, where GQDs generate ROS under light irradiation and NaBH₄ acts as a reducing agent to facilitate the degradation process.

CONCLUSION

In conclusion, the fabrication of graphene quantum dots utilizing glucose as a precursor through the carbonization method was successfully achieved. Characterized by a particle size of 24 nm, the GQDs displayed a spherical morphology and FTIR analysis further validated the existence of relevant functional substituents bonded to the surface of GQDs. The GQDs exhibited strong blue emission under irradiation by UV lamp at 305 nm, exhibiting broad PL at 350 nm upon excitation at 300 nm. This revealed the excitation-dependent PL activity of GQDs. The absorption spectrum indicated the characteristic peak of GQDs with a bandgap energy of 3.81 eV, confirmed by the $n \rightarrow \pi^*$ transition peak of the C=O group at 280 nm. Photocatalytic experiments demonstrated that GQDs acted as an excellent photocatalyst in the availability of NaBH₄, possessing a fast reaction rate of 0.126 min⁻¹ under UV light, leading to the degradation of RhB dye by 86%. In contrast, only 30% of the dye was degraded without the photocatalyst. Under visible light, the presence of both NaBH₄ and GQDs resulted in the degradation of 50% of the dye. Thus, the combination of photocatalytic and chemical reduction processes can lead to the efficient degradation of RhB dye. These findings highlight the potential of GQDs as promising candidates for environmental remediation applications.

APPENDICES

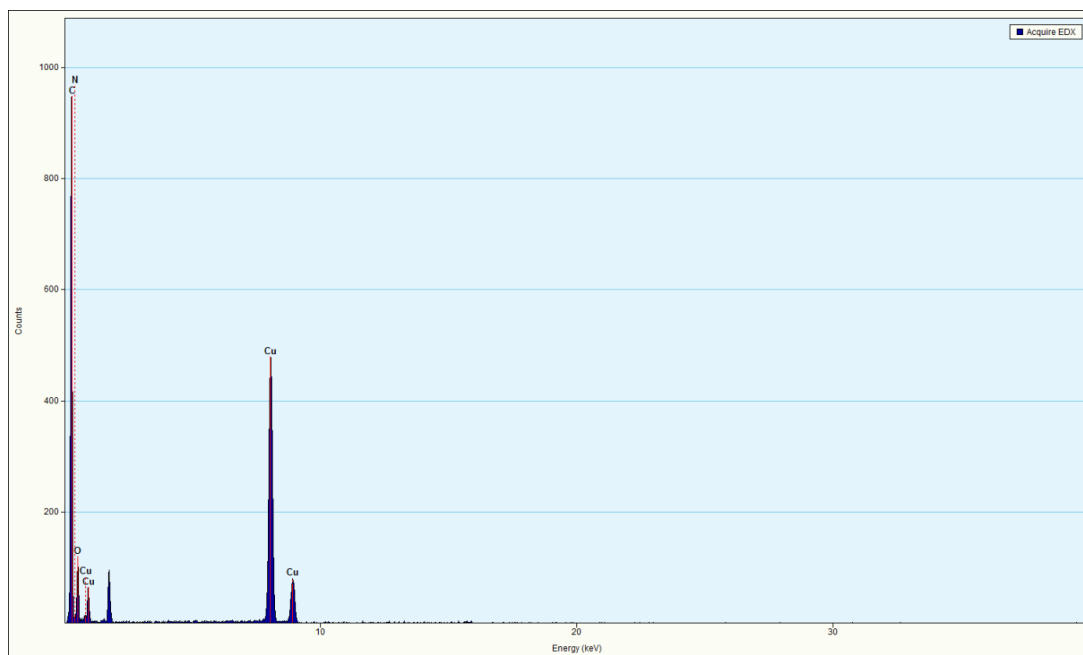



Fig A 1.1 EDX spectrum of graphene quantum dots

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